AEROSPACE REPORT NO.
ATR-97(1091)-2

Stress Measurement Technique Using Microballoons with Carbon Fibers Embedded in an RTV Film

1 June 1997

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Prepared for

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Acknowledgments

This work was in support of the United States Army's Theater High Altitude Area Defense (THAAD) program under the auspices of Dr. Don McClure, THAAD Project Office, Huntsville, Alabama. Funding for the effort was provided by the Ballistic Missile Defense Organization through the POET. The authors are grateful to Dr. Dan Platus for the invaluable technical discussions. They also wish to thank the following personnel for their assistance in carrying out the experiments: George Panos, for setting up and conducting the AE pressure interrogation tests, Pete Valenzuela and Luis Ortega, for preparing the microballoons-filled RTV specimens and conducting mechanical stress tests for AE interrogation.

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1. Background

1.1 Fundamental Theory

Microballoons are tiny, hollow, thin-walled, glass spherical shells in various sizes. Their diameters vary from 1 to 160 μm. They are commercially available in bulk for use as low cost, lightweight filler to be mixed with resins and other materials. Hollow glass microballoons have been used as embedded pressure sensors to record the maximum pressure attained in a fluid. This technique is based on the observation that microballoons with an extensive size distribution exhibit a wide rupture strength distribution (Refs. 1, 2, 3).

Consider a group of hollow glass microballoons of various diameters, mixed with a carrier fluid or grease subjected to an increasing pneumatic pressure. The microballoons in a typical sample will exhibit a random distribution in their rupture strength. The weaker balloons break first at lower pressures, while the stronger balloons survive to higher pressures. As the microballoons break, they generate acoustic emission (AE) that can be readily monitored using standard AE instrumentation. The distribution of rupture strengths can be used to measure the maximum attained pressure in a system by utilizing the Kaiser effect (Ref. 4). The Kaiser effect states that during subsequent pressurization of the microballoons, no AE will be generated until the gas pressure becomes greater than the previously applied unknown pressure, thereby indicating the latter. In the experimental procedure, a sample of the microballoons is exposed to an unknown pressure. The exposed sample is then placed in a pressure chamber and monitored for AE activity as the hydrostatic gas pressure is increased. The pressure at which the AE activity begins is a measure of the maximum pressure seen by the sample. Figure 1 depicts the histogram of the number of AE events from a sample of microballoons that had been previously exposed to a hydrostatic pressure of 100 psi.

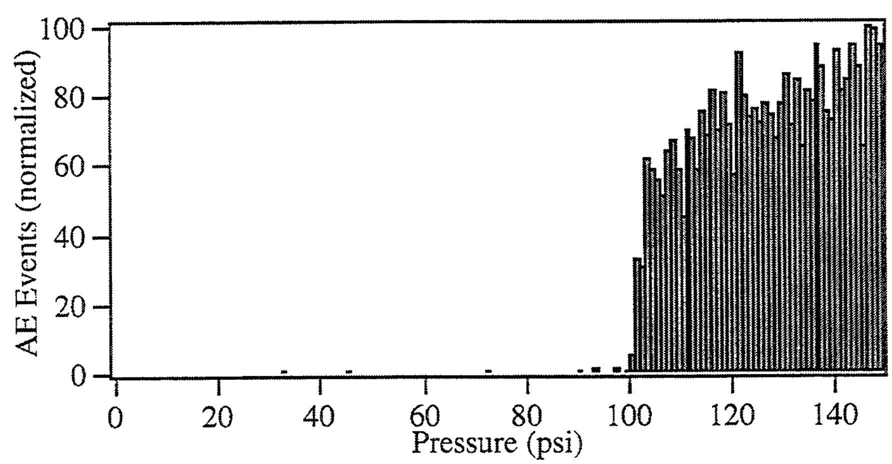

Figure 1. Histogram of the number of AE events from a sample of microballoons that had previously been exposed to 100 psi.

1.2 History of Pressure Memory Measurements Using Microballoons

The pressure memory technique was successfully used to determine the maximum pressure in locations difficult to instrument. For example, in the Titan solid rocket motor (SRM), the technique was used to measure the pressure exerted on the clevis joint by trapped O-ring grease during the stacking operation. Microballoons mixed with grease were also used to record the maximum overpressure levels experienced at various locations of the Titan launch pad (Ref. 2). In both cases, the 3M C15/250 microballoons were mixed with Dow Corning Molykote® 55 grease. The burst pressures of the microballoons chosen for these applications covered a range of 50 psi to more than 1400 psi. The measurement resolution for maximum pressures of approximately 100 psi was better than 5 psi.

2. Stress Measurement Technique Development Using Microballoons

2.1 Motivation

During the assembly of an infrared (IR) sapphire seeker window for the Ballistic Missile Defense Organization (BMDO) Theater High Altitude Area Defense (THAAD) missile system, interest was expressed in measuring the interface compression stress between the window and RTV mounting gaskets. It was estimated that the compression stress associated with the installation was 50 psi or less. Since only the magnitude of the applied torque to each mounting screw was specified, and since the fraction of the applied torque energy going to the screw depended on the magnitude of the friction, the resulting force in each mounting screw could vary significantly.

The measurement of this interface compression was difficult. The measuring sensor must be flexible so that it does not disturb the local stress field. In addition, the device has to be small in dimension: on the order of 4 to 6 mm. Since microballoon-filled sensors could be both small and flexible, an investigation into developing a device using microballoons was undertaken.

2.2 Concept Development

In the early stages of development, the 3M C15/250 microballoon-filled grease was used to fill a tiny hole in a piece of thin RTV simulating the gasket. The RTV pieces containing the grease were then pressurized to generate known magnitudes of compressive stresses. However, when these specimens were interrogated, no conclusion could be drawn from the AE histograms associated with specimens that were stressed at different levels. Also, significant AE signals were recorded from the low applied gas pressure, as if these pieces of RTV were not compressed. It was speculated that this was the result of the following factors:

- Voids existed after the grease was filled into the holes.

- The RTV material was incompressible. In other words, the volume of the hole remained constant whether the specimen was pressed or not. Therefore, the grease in the hole was not subject to any pressure from the wall of the holes.

It was thus decided to abandon the idea of using grease in the gasket. Instead, it was decided that the stress sensor should be very thin, such as the one made from RTV, and should reside at the interface of the two adjacent elements. A detailed description of the specimen configuration is presented in the next section.

3. Experimental Testing and Observation

The experiment was composed of three steps: fabrication of specimens, generation of mechanical stresses, and acoustic interrogation. The second step corresponds to the maximum compression registration or recording. The third step corresponds to the laboratory interrogation. In the real case, the stress sensor will be brought to the pressure chamber to determine the maximum compression that was experienced by the sensor.

3.1 Fabrication of Specimens

3.1.1 RTV with Microballoons

As in the Titan O-ring pressure detection, 3M C15/250 glass microballoons were used. The matrix was GE-RTV-11 with two parts silicone. Typically, 1–2 wt% microballoons (15–30 vol %) was used. A mixture of microballoons in RTV-11 was cured at room temperature. The thickness of the cured RTV sheet was typically 0.13 mm (5 mils). Pieces of 10 mm square specimens were cut for mechanical and AE testing. The mechanical testing will be described in the next section.

3.1.2 Carbon Fiber-Reinforced RTV with Microballoons

An important part of the experiment was to fabricate a specimen that, when subject to uniaxial compression, generated biaxial compression on the microballoons. It was hoped that the biaxially compressed microballoons would buckle in a similar manner as the microballoons did in a gas compressed chamber. In general, the RTV matrix will stretch out laterally when the surface is compressed because of Poisson's effect. In this experiment, there is no lateral direction stress when compression is applied to all three types of specimens. A lateral direction constraint is needed if a lateral direction compression is preferred.

Carbon fibers are known to have high axial stiffness. By using carbon fibers in the RTV matrix, a certain degree of lateral constraint can be achieved. Chopped fibers can be mixed randomly in orientation in the RTV, thus having a transversely isotropic property in the plane of the sheet. Continuously running fibers, on the other hand, will provide maximum constraint in the fiber running directions.

For the experiment, both chopped and continuous running fibers were used. Pan-based T300 carbon fibers were used in both cases. The fabrication of the specimens was similar to that described in Section 3.1.1. When chopped fibers were used, they were added to the RTV, together with the microballoons, before curing. When continuous fibers were used, the carbon fibers were wrapped on two pieces of glasses first (0° and 90° directions, respectively). Microballoon-filled RTV was then poured between two pieces of glasses. The thickness of the chopped fiber and cross-ply fiber-reinforced specimens was 0.25 and 0.89 mm (10 and 35 mils), respectively.

3.2 Generation of Mechanical Stresses

This step in the experiment corresponds to the maximum compression registration or recording onto the stress sensor. The Riehle loading frame was used to apply the compression. Upper and lower cylindrical rods were used as the load-applying fixture. Both the upper and lower loading rods on the frame were 1.0 in. in diameter. To simulate the effect of an RTV gasket, a piece of RTV-11 pad approximately 2.5 cm × 2.5 cm × 2.5 mm thick (1.0 in. × 1.0 in. × 0.1 in thick) was placed between the two loading rods. The specimen, which was smaller than the size of the loading rod, was placed between the RTV pad and the lower loading rod. As the load was applied, the RTV pad was squeezed out in the lateral direction beyond the loading rods. However, the stress on the specimen was simply the load divided by the area of the loading rods.

3.3 Acoustic Interrogation

The system used to interrogate the microballoon sensors consisted of a Physical Acoustic Corporation (PAC) R15 transducer, a PAC 1220 preamplifier, and a PAC 3004 analysis unit. The PAC 3004 is an AE analyzer that provides a convenient method for modifying the signal gain as well as providing some in-line filtering capability. The output from the PAC unit was directed to an HP 3400A root-mean-square (RMS) voltage meter. The RMS voltage provided a convenient measure of the magnitude of the AE activity in the sample. A pressure chamber utilizing N_2 as a pressurant was constructed to interrogate the microballoon samples. The sample was placed on the PAC R15 transducer using a water-based couplant. During the experiment, the chamber was pressurized at a rate of ~ 0.4 psi/sec. The data acquisition was controlled using a LabView program written to capture and display the RMS voltage output. Figure 2 is a typical example of the AE data acquired from the system. The displayed signal was recorded during the testing of a fiber-reinforced RTV sample that had been exposed to a uniaxial compressive stress of 500 psi. The data in Figure 2 dramatically illustrate the differences in the behavior of the microballoons in response to a uniaxial compressive loading. AE activity was limited below 750 psi, then increased rapidly as the pressure was increased, to the maximum chamber pressure of 1400 psi. In a purely hydrostatic measurement, the onset of significant AE would be expected to occur close to 500 psi.

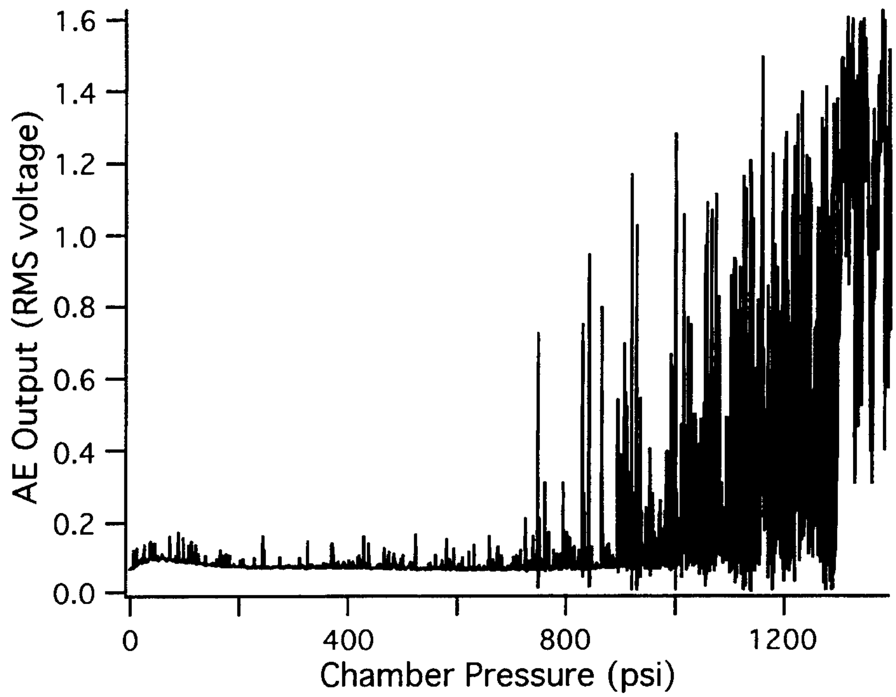

Figure 2. AE data monitored during the hydrostatic pressure loading of a microballoon/RTV sensor. The RTV had been subjected to a uniaxial compressive load of 500 psi.

As discussed in Section 1, the microballoon sensors were developed to monitor hydrostatic pressure conditions in hostile environments. When subjected to pure hydrostatic loading, microballoons provide a very precise marker of the maximum pressure obtained. One of the goals of this study was to extend the utility of the microballoon sensors to uniaxial loading conditions such as those seen at sealing surfaces and O-rings. An array of sensor configurations was examined in an attempt to provide loading conditions that might approximate the hydrostatic loading found in the original microballoon sensors. Of particular interest was an attempt to reinforce the RTV with carbon fibers. It was thought that the Poisson deflections generated by uniaxial loading would be constrained by the carbon fibers. Three carbon fiber configurations were examined in this initial study: cross ply, chopped fibers, and no carbon fiber reinforcement. Samples for each configuration were loaded by uniaxial compression before being subjected to hydrostatic pressure with AE monitoring. The data recorded for each sample were examined to determine the chamber pressure that corresponded to the onset of significant AE activity. The definition of significant AE activity is highly subjective, which contributed to some of the uncertainty in the measurement. Figure 3 is a comparison of the AE signals between samples with cross-ply reinforcement. The sample with no preloading had an AE initiation of ~180 psi, while the sample loaded to 500 psi had AE initiation at ~750 psi.

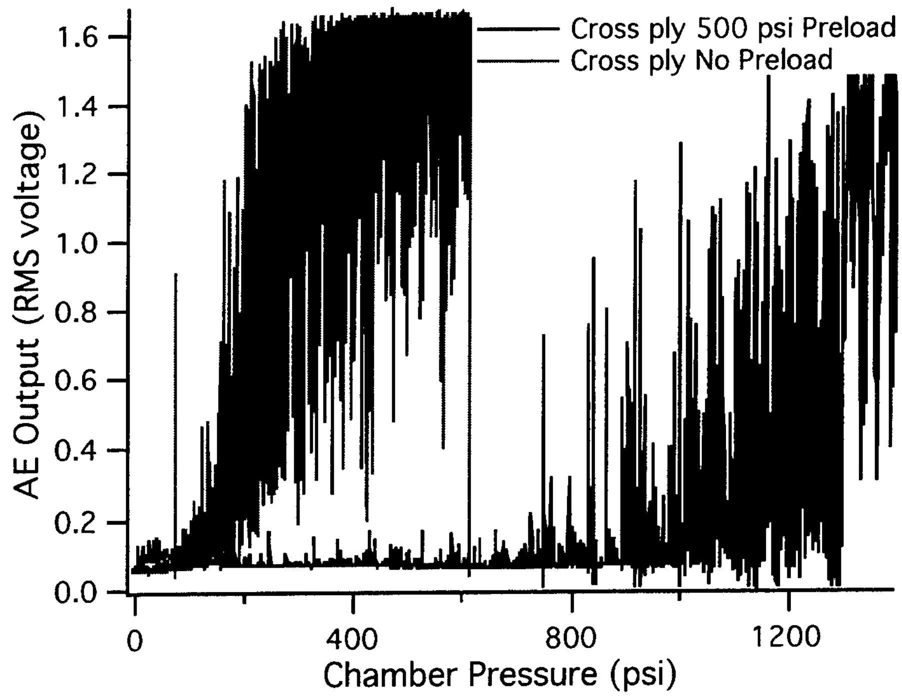

Figure 3. Comparison of AE signal between cross-ply samples–one exposed to no preload and the other to a preload of 500 psi.

Figure 4 shows the AE initiation pressure plotted against the uniaxial preload for each of the carbon fiber reinforcement configurations. At least two observations can be made from the data compiled in Figure 4. First, over the limited pressure range evaluated during this investigation, the response of the microballoon sensors appears to be linear. Second, the effect of the fiber reinforcement tends to increase the slope of the curve and decrease the data scatter, improving sensitivity of the technique. Based on these data, the slope of the AE initiation pressure vs the preload stress ranges between 1.3 and 1.6. The specimens of cross-ply reinforced fibers has the highest slope values.

The data presented in Figure 4 shows clearly that even with uniaxial loading, the microballoons provide a marker of the maximum pressure obtained. However, because of the variability in the data, the pressure resolution of the technique as it is applied here is limited to approximately 100 psi.

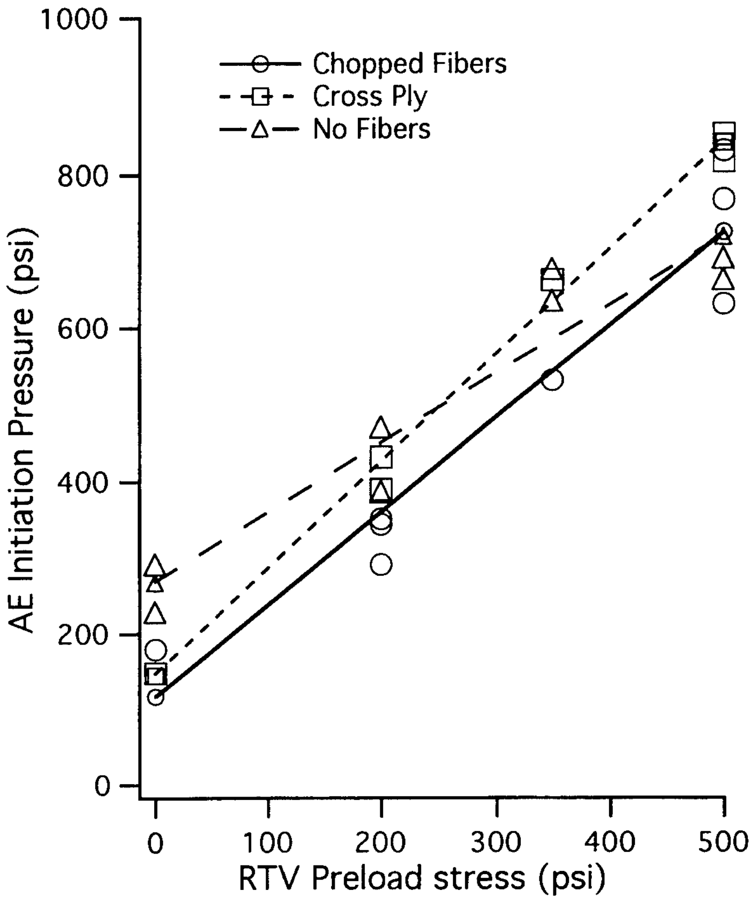

Figure 4. Comparison of the AE initiation pressure for the different microballoon sensor configurations and initial load exposures.

4. Analysis

4.1 Buckling Analysis of a Spherical Thin Shell

A buckling analysis was performed for a thin wall spherical shell to determine the critical external pressure at which fracture of the spherical shell occurs as a result of buckling. The purpose of the buckling analysis was to estimate the external pressure distribution around the microballoons in embedded condition when the specimens are loaded uniaxially under compression. It is understood that the microballoons do not necessarily have uniform thin wall configuration. However, it is felt that such an assumption can statistically catch the essence of the microballoon behavior in calculating the rupture strength.

Two pressure cases were analyzed in the buckling: case 1, with a uniform external pressure of intensity p_o, and case 2, with a pressure intensity distribution $p'_o \cos^2(\varphi)$, where φ is the angle measured from the pole.

The results indicate that the case 2 maximum peak pressure p'_o at buckling is about 64% that of the critical p_o for the uniform pressure case (case 1). This ratio agrees with the slope of 1.6 estimated for specimens with cross-ply carbon fiber reinforcements. In other words, for the specimens that have cross-ply carbon fibers, the pressure distribution around the microballoons can be represented by $p'_o \cos^2(\varphi)$.

4.2 Comparison of Analytical Prediction with Experimental Results

In Figure 4, it is observed that the interrogation gas pressure level at which AE signals initiate is always higher than the mechanically applied average compressive stress. This observation suggests that the actual pressure intensity distribution of microballoons embedded in the RTV is of the form $p'_2 \cos^2(\varphi)$. Further, the specimens with cross-ply fiber reinforcement tend to have higher slope, meaning that the pressure intensity distributions for these specimens are more uniform than distributions for the unreinforced specimens.

5. Summary and Conclusions

This investigation examined the use of microballoon impregnated RTV to determine the maximum compressive stress of the RTV. The use of microballoon-impregnated RTV is an extension of microballoon sensors previously developed to measure the hydrostatic pressure in remote and hazardous locations. The results of this study showed that the microballoons, when embedded in RTV thin films, can provide an indication of the maximum applied compressive stress. The use of carbon fibers appears to provide some lateral constraints and thus increase the repeatability of the test data. However, the resolution or accuracy will be much higher than the 5 psi resolution found in the hydrostatic case. A further investigation of the specimen configurations–such as types of microballoons, casting matrix, and lateral restraining technique–to enhance the resolution is needed. Another potential line of investigation that could provide better resolution would be to replace the hydrostatic pressure chamber with a uniaxial loading frame. The use of a loading frame would provide a truer comparison between the load applied during the measurement and the load applied during the sample interrogation. While the use of microballoon-filled RTV did not satisfy the requirements of the IR window measurement, it does have potential applications in the measurement of other more highly loaded joints.

7. References

1. G. F. Hawkins, J. R. Lhota, J. R. Hribar and E. C. Johnson, "Acoustic Emissions from Pressurized Microballoons," *Review of Progress in Quantitative NDE*, Vol. 12, ed. D. O. Thompson and D. E. Chimenti (Plenum Press, New York, 1993), pp. 989.

2. J. R. Lhota, P. M. Sheaffer, and G. F. Hawkins, "Smart Materials Used in Frequency Selective Passive Sensors", *Review of Progress in Quantitative NDE*, Vol. 11A, ed. D. O. Thompson and D. E. Chimenti (Plenum Press, New York, 1992), pp. 1097–1102.

3. J. R. Lhota, G. C. Panos, G. F. Hawkins, and J. N. Schurr, "Pressure Measurement Using Microballoons," *Review of Progress in Quantitative NDE*, Vol. 7, ed. D. O. Thompson and D. E. Chimenti (Plenum Press, New York, 1988), pp. 691–695.

4. J. Kaiser, *Untersuchugen uber das Auftreten von Gerauschen beim Zugversuch*, Ph.D. thesis (Technische Hochschule, Munich, 1950).